

of the amorphous carbon and crystalline graphene residue, which is lightest in weight of the chamber contents and has weak magnetic properties.

**[0069]** The third phase with its debundled carbon nanotubes is then separated from the other phases. One particularly useful technique for accomplishing this separation is to freeze the contents of apparatus **70**, using an ultra-cold freezer ( $-80^{\circ}\text{C.}$ ) or submersion in dry ice pellets, then removal of the frozen contents from the cylindrical outer body **72** by driving plunger **76** upward after the end closure **78** has been removed.

**[0070]** The plunger **76** may be made of, for example, high density polyethylene. A cutting or other separating operation may be conducted on the frozen contents to collect the CNT-fraction frozen layer. This cutting or separation operation should be carried out immediately, before thaw is allowed to occur. It has been found that the phases will peel away from one another without requiring a cutting instrument or excessive physical force.

**[0071]** All single-wall carbon nanotubes can be categorized as metallic semi-metals, or semiconducting depending on their conformation. It has been found that one of the potential benefits of the magnetic phase separation step **12** is that the recovered CNT layer has in some cases stratified into a sub-layer of CNTs having metallic properties ("metallic CNTs") and a sublayer of CNTs having semiconductive properties ("semiconductor CNTs"). The latter semiconductor CNTs are particularly desirable for many applications, and are difficult to isolate from the metallic nanotubes using conventional processes.

**[0072]** As described above, a stabilizing dispersant (e.g., sodium dodecyl sulfate (SDS)) bonds to the CNT, presumably via van der Waal forces, and/or creates a micelle structure to establish a protective coating around the CNTs. The anionic charge of the SDS facilitates an electrophoresis operation in stage **14** of FIG. **1** for reducing the content of amorphous carbon in the recovered, debundled CNT fraction, as described below. An embodiment of an apparatus for conducting the colloidal electrophoresis step **14** is illustrated in FIG. **6**.

**[0073]** Referring to FIG. **6**, colloidal electrophoresis apparatus **80** includes a cylindrical housing **82** having end closures **83** and **84** at its opposite ends. The housing **82** and end closures **83**, **84** may be made of any suitable material, such as polycarbonate, glass, ceramic, or other materials. End closure **83** includes a swivel joint **85** and a positive electrode centered in the swivel joint **85**. Similarly, end closure **84** includes a swivel joint **86** with a negative electrode centered in the swivel joint **86**. Although not shown, a rotary system is included to rotate the cylindrical housing **82** about its longitudinal axis. Suitable bearing members may be included for facilitating rotation of the swivel joints **85**, **86** relative to the housing **82**.

**[0074]** A filter cartridge generally designated by reference numeral **90** is received in the cylindrical housing **82** and generally centered along the length of the housing **82**. The filter cartridge **90** is fixedly yet removably attachable to the housing **82** to allow the cartridge **90** to rotate with the housing **82**.

**[0075]** The filter cartridge **90** includes an annular membrane mount **91** for retaining a plurality of filter cassettes **92-94**. FIG. **7A** shows an end view of the filter membrane cassette **92**, which includes an outer rim **92a** having screw holes **92b**, concentric first and second O-rings **92c**, **92d**, and a

filter membrane **92e**. As best shown in FIG. **7B**, the outer edge of filter membrane **92e** passes serpentine-like through mounted are thereby mounted to the O-rings **92c**, **92d**. Filter cassette **92** optionally also contains access ports **92f**, **92g** for reasons discussed below. Filter cassettes **93**, **94** may be constructed similarly to filter cassette **92**.

**[0076]** Upstream (to the right in FIG. **6**) from the filter cartridge **90** and the first filter membrane cassette **92** is a first zone or compartment **95**. Between the first and second filter cassettes **92**, **93** is a second zone or compartment **96** contained within the filter cartridge **90**. A third zone or compartment **97** also contained in the filter cartridge **90** is interposed between the second and third filter cassettes **93**, **94**. A fourth zone or compartment **98** is positioned downstream from the third filter cassette **94** and the filter cartridge **90**. The filter cartridge **90** may further include spacer rings between the filter cassettes **92-94**, and fasteners (e.g., bolts) for securing the filter cassettes **92-94** to the membrane mount **91**. It should be understood that the filter cartridge **90** may contain additional filters to subdivide the chamber of the filter cartridge **90** into more zones or compartments.

**[0077]** First, second, third, and fourth entry/drainage/venting ports **95a**, **96a**, **97a**, **98a** extend through the cylindrical housing **82** to zones **95-98**, respectively, for permitting the introduction, venting, and removal of material from zones **95-98**. Although not shown, each port **95a-98a** is provided with a removable cap for establishing a fluid-tight seal. If the cartridge cassettes **92-94** are placed so closely together so as not to create spacing for aligning the entry/drainage/venting ports **96a**, **97a** with zones **96**, **97** directly, then the access ports **92f**, **92g** of the filter cassettes may be aligned with the ports **96a**, **97a** for material introduction/removal and venting. The spacing between cartridge cassettes **92-94** may be particularly limited in instances in which the filter cartridge **90** includes additional (e.g., more than three) cartridge cassettes.

**[0078]** In accordance with an experimental embodiment, zones **95-98** are partially filled through their respective ports **95a-98a** with an aqueous buffer comprised of 25 mM Trizma™ base (Sigma-Aldrich, St. Louis, Mo.); 190 mM glycine, pH 8.3 which increases the mobility of nanoparticles such as CNTs and amorphous carbon during electrophoresis. An exemplary buffer for this stage is tris-glycine. Zone **96** serves as a loading zone to receive the CNT fraction obtained from stage **12**. Amorphous carbon not earlier separated will typically be included in the CNT fraction loaded into zone **96**.

**[0079]** The negative charge of the SDS joined to and/or encasing the CNTs and amorphous carbon is attracted to the positive electrode **87** and creates a particle flow (or migration) in the direction of arrows **99**. The pores in the filter membranes (e.g., **92c**) of filter cassettes **92** and **93** are sized to permit the passage of amorphous carbon, but to intercept and block the passage of the CNTs. The pores of the filter membranes (e.g., **92e**) may be, for example, on the order of about 0.1 nm to about 10 nm for carrying out dialysis. Filter cassette **94** may have a filter membrane with an even smaller pore size. In the event that additional filter cassettes are included in the cartridge **90**, the pore size of the filter membranes preferably becomes incrementally smaller in a downstream direction, i.e., the direction in which arrows **99** point. The use of multiple membrane sizes allows for further size fractionalization of the CNTs. The first and fourth zones **95**, **98** will contain water, buffer, and amorphous carbon, and will be mostly free of the CNTs introduced into compartment **96**.